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METHODS FOR IDENTIFICATION OF APPLICATION CLASSES OF DYES ON TEXTILE MATERIALS

PART III MAN-MADE FIBRES

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110001

April 1975

Indian Standard

METHODS FOR IDENTIFICATION OF APPLICATION CLASSES OF DYES ON TEXTILE MATERIALS

PART III MAN-MADE FIBRES

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Indian Standard

METHODS FOR IDENTIFICATION OF APPLICATION CLASSES OF DYES ON TEXTILE MATERIALS

PART III MAN-MADE FIBRES

0. FOREWORD

0.1 This Indian Standard (Part III) was adopted by the Indian Standards Institution on 20 February 1973, after the draft finalized by the Dyestuffs Sectional Committee had been approved by the Textile Division Council.

0.2 This standard is one of a series of standards on methods of test for identification of application classes of dyes on textile materials. Parts I and II of this standard deal with identification of application classes of dyes on cotton and other cellulosic fibres; and wool, silk and other protein fibres respectively.

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1. SCOPE

1.1 This standard (Part III) prescribes methods for identification of application classes of dyes on man-made fibres, such as secondary acetate, triacetate, acrylic, polyester, polyamide, polyvinyl alcohol, polyvinyl chloride, polyvinyl acetate, polyurethane and polyolefin fibres; their blends with each otner and with natural and regenerated-cellulosic fibres.

1.1.1 The standard is not applicable to protein fibres or blends thereof.

1.2 The methods are applicable to types of dyes normally used for dyeing and printing man-made fibres.

2. PREPARATION OF TEST SPECIMEN

2.1 If the sample under test is in the form of fibres, take a tuft of fibre.

2.2 If the sample is in the form of yarn, take a bundle of yarn about 3 cm in length.

2.3 If the sample is in the form of fabric, take a 3×3 cm test specimen.

NOTE — In case of multi-coloured fabric the specimens shall be taken from different coloured portions of the sample and the different coloured fibres present therein shall be identified separately for their respective classes of dyes.

2.4 Any finish present in the sample shall be removed prior to identification of application classes of dyes by the procedure given in **2.4.1** to **2.4.6**. If the extract is appreciably coloured at any stage it should be analysed individually for the application classes of dyes as given in Appendix A.

NOTE— These procedures are given only as guide and it must be stressed that a number of finishes are likely to be encountered which will not be removed by these treatments and for which certain other treatments may be necessary.

2.4.1 Treat the specimen with 1 g/l of a non-ionic detergent at 60 to 70°C for 15 to 20 minutes. Wash well first with warm and then with cold water and dry.

2.4.2 Boil the specimen obtained in **2.4.1** with 50 ml of carbon tetrachloride under reflux for 5 minutes.

2.4.3 Boil the specimen obtained in **2.4.2** with 50 ml of ethyl alcohol, under reflux, for 5 minutes.

2.4.4 Boil the specimen obtained in **2.4.3** with 50 ml of distilled water, under reflux, for 5 minutes.

2.4.5 Boil the specimen obtained in **2.4.4** with dioxane, under reflux, for 5 minutes.

2.4.6 Boil the specimen obtained in **2.4.5** with 50 ml of distilled water containing 2 ml of concentrated hydrochloric acid, under reflux, for 5 minutes.

3. APPARATUS, MATERIALS AND REAGENTS

3.1 Apparatus

3.1.1 *Microscope*

3.1.2 *Test Tubes*

3.1.3 *Separating Funnels*

3.1.4 *Porcelain Crucible*

3.2 Materials

3.2.1 *Lead Acetate Paper*

3.2.2 *Mordanted Wool*

3.2.3 *Scoured Acetate Fabric*

3.2.4 *Scoured Cotton*

3.2.5 *Scoured Wool*

3.2.6 *Magnesium Ribbon*

3.2.7 *Zinc Dust, Pure*

3.3 Reagents

3.3.0 *Quality of Reagents* — Unless specified otherwise, pure chemicals shall be employed in tests and distilled water (*see* IS : 1070-1960*) shall be used where the use of water as reagent is intended.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the test results.

3.3.1 *Acetic Acid* — (a) 5 percent $\left(\frac{m}{v}\right)$, and (b) 30 percent $\left(\frac{m}{v}\right)$.

3.3.2 *Ammonia Solution* — (a) 1 percent $\left(\frac{v}{v}\right)$, and (b) concentrated (sp gr 0·88).

3.3.3 *Carbazol* — 0·1 percent.

3.3.4 *Chromotropic Acid Solution* — 5 percent (aqueous).

3.3.5 *Ether* — *See* IS : 336-1964†.

3.3.6 *Ethylene Diamine Hydrate* — sp gr 0·960.

3.3.7 *Ethylene Diamine Tetra-acetic Acid Disodium Salt*

3.3.8 *Formic Acid* — 85 percent.

3.3.9 *Formosul G*

3.3.10 *Glycerol*

3.3.11 *Hydrochloric Acid* — (a) 16 percent $\left(\frac{v}{v}\right)$, and (b) concentrated (sp gr 1·18).

3.3.12 *Hydrogen Peroxide* — 30 percent $\left(\frac{m}{v}\right)$ (100 volumes).

3.3.13 *Nitric Acid* — Concentrated.

3.3.14 *Non-ionic Detergent*

3.3.15 *O-Chlorophenol*

3.3.16 *Paraffin, Liquid*

3.3.17 *Pyridine*

3.3.18 *Sodium Carbonate*

3.3.19 *Sodium Hydroxide Solution* — (a) 5 percent, and (b) 20 percent.

3.3.20 *Sodium Hydrosulphite* — (a) 0·2 percent $\left(\frac{m}{v}\right)$ solution, and (b) solid (*see* IS : 1919-1961‡).

*Specification for water, distilled quality (*revised*).
†Specification for ether (*revised*).
‡Specification for sodium hydrosulphite, technical.

3.3.21 *Sodium Hypochlorite Solution* — containing 2 g/l of available chlorine.

3.3.22 *Sodium Nitrate*

3.3.23 *Sodium Sulphate Solution* — 0·2 percent $\left(\frac{m}{v}\right)$.

3.3.24 *Solution of Dispersing Agent* — 10 percent $\left(\frac{m}{v}\right)$.

3.3.25 *Sulphuric Acid* — (a) 5 percent $\left(\frac{v}{v}\right)$, and (b) concentrated.

3.3.26 *Tannic Acid*

3.3.27 *Toluene*

4. PROCEDURE

4.1 Microscopic Examination — Examine the test specimen under the microscope. If the dye is found to be present on the surface of the fibre as particles, it indicates pigment dyes, namely, carbon black, vat, azoic or phythalocyanine [*see* 3 (b) under Additional Tests in Appendix A].

4.2 For identification of application classes of dyes, follow the procedure as given in Appendix A.

NOTE 1 — While identifying the dyes used for dyeing pale shades, it is advisable to use large specimens and large quantities of reagents and concentrate the extract before making the tests.

NOTE 2 — Before identification the fibres in the blend may be separated, by a suitable method, if necessary.

APPENDIX A

(*Clause* 4.2)

IDENTIFICATION OF APPLICATION CLASSES OF DYES ON MAN-MADE FIBRES

TEST SPECIMEN

FIRST PORTION

Boil about 0·5 g of test specimen in 1 percent ammonia solution for one minute

A considerable amount of colour bleeds into the solution

Discard the specimen. Acidify the solution with 30 percent acetic acid. Add 0·5 g of scoured wool and dye for 5 to 10 minutes at 80 to 90°C

If the wool is dyed, it indicates ACID DYE

Slight or no colour bleeds into solution
Boil 0·5 g of fresh test specimen with 5 percent acetic acid for one minute

The solution is distinctly coloured

Divide the solution into two parts:

a) To one part add tannic acid
Formation of precipitate indicates BASIC OR MODIFIED BASIC DYE

b) To the other part add scoured wool and warm for 4 to 5 minutes
Staining of wool indicates BASIC OR MODIFIED BASIC DYE

The solution is not coloured

Boil 0·5 g of fresh test specimen with 5 percent sodium hydroxide solution for 1 to 2 minutes

If considerable amount of colour is stripped, divide the solution into two parts:

a) To one part add scoured cotton and warm for 5 to 10 minutes. Deep staining indicates DIRECT DYE. A little or no staining indicates REACTIVE DYE

b) To another part add formosul G. The solution is decolourised and original colour is not restored. DIRECT DYE is confirmed. (Non-dischargeable direct dyes will not respond to this treatment).

NOTE — For determining whether the dyeing has been after-treated with formaldehyde or copper, chromium or nickel proceed as follows:

a) *Direct Dyes after-treated with Formaldehyde* — Warm 0·5 g of original test specimen (that is, before definishing) in 5 percent sulphuric acid. Allow it to cool. Discard the sample and test as in (i) or (ii):

i) Add 0·1 percent of carbazol dissolved in concentrated sulphuric acid to the solution drop by drop.
The formation of a blue colour precipitate indicates the presence of FORMALDEHYDE

OR

ii) Add 1 ml concentrated sulphuric acid and 1 ml of 5 percent aqueous chromotropic acid solution to the solution
The formation of violet or red-violet colour either immediately or warming at 60 to 70°C for 5 to 10 minutes indicates presence of FORMALDEHYDE

b) *Direct Dyes After-Treated with Copper, Nickel or Chromium* — Take 1 ml of 100 volume hydrogen peroxide in a watch glass and add 2 to 3 drops of concentrated ammonia solution. After the evolution of bubbles has ceased, add about 0·1 g of fresh test specimen to it.
Any vigorous action indicates the presence of COPPER, NICKEL OR CHROMIUM

SECOND PORTION

Boil 0·5 g of test specimen in glacial acetic acid for 5 minutes

a) *If some of the colour is stripped*, cool, add ether to the solution and shake well. If the ether layer is coloured, it indicates DISPERSE DYE

b) Treat a fresh specimen in hot liquid paraffin at 100°C for 5 minutes. If the colour is stripped add scoured acetate fabric
Tinting of the acetate fabric confirms DISPERSE DYE

THIRD PORTION

Reflux 0·5 g of fresh test specimen with dioxane-water mixture in the proportion of 1 : 5 or 1 : 10 for 2 to 3 hours. If the dioxane extract is coloured, take 20 ml of the extract and add to it 5 to 10 ml of 20 percent sodium hydroxide and 10 to 15 mg of formosul G and boil for 5 to 10 minutes. Note any change in colour. Then add to it 1 to 2 ml of 100 volume hydrogen peroxide. If the original colour of the solution is now restored, it indicates VAT DYE (the exceptions are with certain blue dyes, the leuco compounds, which are also blue in colour). If not, it indicates AZOIC OR CERTAIN DIAZOTISABLE AZO DYES (direct dye, diazotised and developed; and developed disperse dyes)

Boil 0·5 g of fresh test specimen with 5 to 10 ml of 20 percent sodium hydroxide solution for 2 minutes. Add 4 to 5 ml of water and 15 to 45 mg of solid sodium hydrosulphite and boil for 1 minute

(a)

If the colour of the test specimen is destroyed or changed to another tone and is restored on exposure to air, it indicates SULPHUR OR SULPHURIZED VAT DYE

Boil a fresh test specimen with 10 percent Hydrochloric acid for 30 seconds. Cool and add 3 mg of Magnesium ribbon or pure zinc dust and warm for 2 to 3 minutes. Place lead-acetate paper on the mouth of the test tube

If lead acetate paper turns black and the vapours give smell of hydrogen sulphide gas, SULPHUR OR SULPHURIZED VAT DYE is confirmed

Treat a fresh test specimen with cold dilute hypochlorite solution for 5 to 10 minutes

If the specimen becomes colourless, SULPHUR DYE is confirmed

(b)

If the colour of the test specimen readily becomes brown and changes to black when exposed to air, it indicates ANILINE BLACK

Warm a fresh test specimen with concentrated sulphuric acid for a few seconds and pour the liquid to little cold water

If a dull green colour is produced, ANILINE BLACK is confirmed

(c)

If the colour of the test specimen is reduced to another tone and on exposure to air original colour is restored, it indicates a VAT OR ANTHRAQUINONE REACTIVE DYE

NOTE — The exceptions are with certain blue dyestuffs, where the colour of the reduced vat dye or leuco compound is also blue like the original dyestuff

Treat a fresh test specimen with cold dilute sodium hypochlorite solution for 5 to 10 minutes

If the test specimen is decolourized or its colour becomes light in tone and the original tone is not restored on treatment with 0·2 percent sodium hydro-sulphite solution in cold for 2 minutes, it indicates ANTHRAQUINONE REACTIVE DYE; if not, it indicates VAT-DYE

(d)

If the test specimen is permanently decolourized or is changed to another tone and does not come back to the original colour on oxidation, it indicates AZOIC OR CERTAIN DIAZOTISABLE AZO DYES OR NON-ANTHRAQUINONE REACTIVE DYES

1) *Azoic and Certain Diazotisable Azo Dyes* —
Boil 0·5 g of fresh test specimen with 5 ml of Pyridine for 1 to 2 minutes
Protuse bleeding of colour indicates AZOIC DYE
Slight bleeding of colour indicates DEVELOPED DISPERSE DYE
No bleeding of colour indicates DIRECT DYE (diazotised and developed)
Treat 0·5 g of fresh test specimen with non-ionic detergent at 85 to 90°C for 2 to 3 minutes. Discard the test specimen, add 0·5 g of scoured cotton to the solution and boil for another 2 to 3 minutes
If the cotton is stained, DIRECT DYE (*diazotised and developed*) is confirmed. If there is no staining of the cotton, DEVELOPED DISPERSE DYE is confirmed

2) *Reactive Dyes* — Reflux 0·5 g of fresh test specimen with a solution containing 1 ml of concentrated sulphuric acid and 2 g/l sodium sulphate diluted to 1 litre with water for 15 minutes. If colour bleeds into solution, add scoured wool. If wool is stained, it indicates REACTIVE DYE of the heterocyclic halogenated type
No bleeding of colour indicates REACTIVE DYES of types other than the heterocyclic halogenated

(e)

If the colour of the test specimen does not change, it indicates CHROME DYE

Warm the test specimen with 16 percent Hydrochloric acid for 1 to 2 minutes. Cool and add 57 : 43 Pyridine water. If colour bleeds into solution, add mordanted wool to the solution and warm for 2 to 3 minutes

If the wool is dyed, it indicates CHROME DYE

FOURTH PORTION

Treat 0·5g of fresh test specimen with 57 : 43 Pyridine-water in cold for 5 to 10 minutes

If the liquor is stained, it indicates SOLUBLE DISPERSE DYE

FIFTH PORTION

Dissolve 4 g of Ethylene diamine tetra-acetic disodium salt in 100 g of Glycerol. Heat the test specimen in this mixture at 140°C

Observe change in colour of the specimen after 1 to 2 minutes and after 20 minutes

If the colour of the specimen changes in 1 to 2 minutes, it indicates 1 : 1 METAL COMPLEX DYE

If the colour of the specimen changes after 20 minutes, it indicates 1 : 2 METAL COMPLEX DYE

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ADDITIONAL TESTS

1) *Extraction Test* — Extract 0·5 g of fresh test specimen with 15 ml of 57 : 43 pyridine-water in a test tube by keeping it in a beaker of boiling water for 10 to 15 minutes, or until sufficient amount of colour bleeds into the reagent. Discard the test specimen and note the colour of the extract. Pour the solution into a separating funnel, add 15 ml of toluene, shake well and allow the two layers to separate. The distribution of dyes between the two layers is as follows:

Toluene Layer	*Pyridine-Water Layer*
All disperse dyes	All direct dyes
Some neutral dyeing metallized dyes (1 : 2 metal complex dyes)	All basic dyes
Some vat dyes	All acid dyes
Some reactive disperse dyes	All acid dyeing metallized dyes (1 : 1 metal complex dyes)
All azoic combinations	All chrome dyes
	Logwood:
	Some neutral dyeing metallized dyes (1 : 2 metal complex dyes)

If the toluene layer is coloured, wash it with water thrice. Separate the toluene layer again and evaporate it. Disperse the residue with a few drops of 10 percent solution of a dispersing agent in water. Add scoured wool and scoured acetate fabric to this and warm for 15 minutes.

If only wool is dyed, it indicates NEUTRAL DYEING METALLIZED DYE (that is, 1 : 2 METAL COMPLEX DYE).

If both wool and acetate fabric are dyed, it indicates DISPERSE DYE.

If the pyridine-water layer is coloured dark cherry-red, it indicates *Logwood.* Add 1 to 2 ml of concentrated hydrochloric acid, it turns yellowish brown; shake with toluene, the colour remains in pyridine-water layer.

NOTE — In case of Chrome Dyes pyridine-water layer is coloured. But sometimes the toluene layer is also stained to a different colouration than original dyeing.

2) *Ash Test* — Ash 0·2 to 0·3 g of fresh test specimen in a porcelain crucible. Add 0·2 to 0·3 g of flux composed of equal parts by weight of powdered sodium carbonate and sodium nitrate. Fuse the mixture and allow it to cool. The presence of any metals is indicated by the colour of the fused mass as follows:

Colour of Fused Mass	*Metal Present*
Yellow Colour	Chromium
Royal Blue	Cobalt
Faint Blue-Green	Copper
Blue-Green	Manganese
Brown	Nickel

The presence of cobalt or manganese indicates NEUTRAL DYEING (1 : 2 METAL COMPLEX DYES).

The presence of chromium indicates DIRECT DYE after-treated with chromium salt, chrome dyes or metallized dyes (that is, 1 : 1 metal complex dyes and 1 : 2 metal complex dye).

The presence of copper or nickel indicates DIRECT DYE after-treated with copper or nickel salt respectively.

3) *Miscellaneous Tests*

a) *Test for Reactive Disperse Dye on Nylon 6 and 66* — Dissolve 0·5 g of fresh test specimen in formic acid or o-chlorophenol and pour the resulting solution into 1 ml of ethylene

diamine hydrate diluted with 5 to 10 ml of water. Warm for 5 to 10 minutes and then filter. If the dye remains along with the precipitate, it is a REACTIVE DISPERSE DYE.

b) *Test for Pigment Dye* — If pigment dye is found to be present by the microscopic examination (*see* **4.1**) and azoic and vat pigments are found to be absent by the relevant subsequent tests, the pigment dye present may be carbon black or phythalocyanine pigment.
Treat a test specimen with sodium hydroxide and sodium hydrosulphite solution, no discolouration of the specimen indicates CARBON BLACK.
Spot a test specimen with concentrated nitric acid, appearance of bright-green tone indicates PHTHALOCYANINE PIGMENT.

INDIAN STANDARDS

ON

METHODS OF TEST FOR DYES

IS:

1688-1960 Procedure for determination of fastness of dyestuffs

1962-1961 Method for determination of fastness of dyestuffs to metals in the dyebath : chromium salts

1968-1961 Method for determination of fastness of dyestuffs to metals in the dyebath : iron and copper

3859-1966 Method for determination of strength of water soluble azo dyes by reduction with titanium trichloride

4394-1967 Method for evaluating strength of homogeneous vat dyestuffs

4459-1967 Method for determination of strength of direct dyestuffs by dyeing test

4471-1967 Methods for determination of strength of naphthols (azoic coupling components) (gravimetric and volumetric methods)

4472 (Part I)-1967 Methods for identification of the application classes of dyes on textile materials: Part I Cotton and other cellulosic fibres

4472 (Part II)-1968 Methods for identification of application classes of dyes on textile materials: Part II Wool, silk and other protein fibres

4946-1968 Method for evaluation of strength and shade of naphthol

5970-1970 Method for estimation of strength (vat content) of solubilized vat dyestuffs

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